

## PATENT ABSTRACTS OF JAPAN

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(71)Applicant : NIPPON NOHYAKU CO LTD

(22)Date of filing : 09.03.1992

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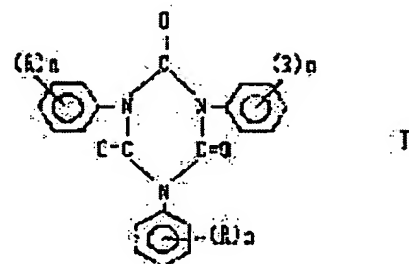
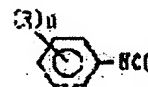
YABUTANI KUNIHIRO

## (54) PRODUCTION OF TRIAZINETRIONES

## (57)Abstract:

PURPOSE: To obtain the subject compound useful as an intermediate for resins by trimerizing an isocyanic ester in the presence of a phase transfer catalyst.

CONSTITUTION: An isocyanic ester of formula I (R is halogen, nitro, hydroxyethyl, etc.) is trimerized in the presence of a phase transfer catalyst (e.g. tetrabutylammonium chloride) to give the objective compound of formula II. The reaction is preferably carried out by using 0.01-0.1mol based on the compound of formula I at 50-200°C. The reaction time is preferably several minutes to 48 hours. When phenyl isocyanate is used as the compound of formula I, 1,3,5-triphenyl-perhydro-1,3,5-triazine-2,4,6-trione is obtained as the objective compound of formula II.



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application converted registration]  
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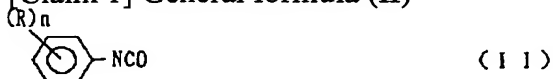
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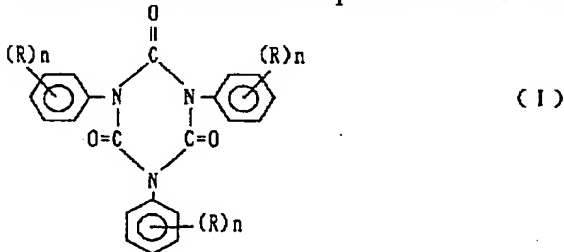
## CLAIMS

**[Claim(s)]**

[Claim 1] General formula (II)



the inside of a formula and R are the same -- or -- you may differ -- a halogen atom, a nitro group, and hydroxyl -- a low-grade alkyl group and low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, A low-grade alkylthio group and low-grade halo alkylthio group, a low-grade alkyl sulfonyl group, A low-grade halo alkyl sulfonyl group, a carboxy group, a low-grade alkoxy carbonyl group, Or you may differ. a low-grade alkylamino radical, a low-grade dialkylamino radical, and a phenyl group -- the same -- A halogen atom, low-grade alkyl group, and low-grade halo alkyl group, a lower alkoxy group, The phenyl group which has 1-5 substituents chosen from a low-grade haloalkoxy radical, low-grade alkylthio group, and low-grade halo alkylthio group, a phenoxy group -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The phenoxy group which has 1-5 substituents chosen from a low-grade halo alkylthio group, a phenylthio radical -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The phenylthio radical which has 1-5 substituents chosen from a low-grade halo alkylthio group, a phenyl sulfonyl group -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The phenyl sulfonyl group which has 1-5 substituents chosen from a low-grade halo alkylthio group, benzoyl -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The benzoyl which has 1-5 substituents chosen from a low-grade halo alkylthio group, an aralkyl radical -- or the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- the aralkyl radical which has 1-5 substituents chosen from a low-grade halo alkyl group, lower alkoxy group, low-grade haloalkoxy radical, low-grade alkylthio group, and low-grade halo alkylthio group is shown, and n shows the integer of 0-5. The general formula characterized by quantifying the isocyanic ester expressed three times under existence of phase transfer catalysis (I)



(-- R and n are the same as the above among a formula.) -- manufacturing method of triazine trione expressed.

[Claim 2] The manufacturing method of the triazine trione given in the 1st term of a claim given phase transfer catalyses are quarternary ammonium salt, phosphonate, or crown ether.

[Claim 3] The manufacturing method of the triazine trione given in the 2nd term of a claim given phase transfer catalyses are quarternary ammonium salt.

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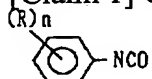
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 CLAIMS
 

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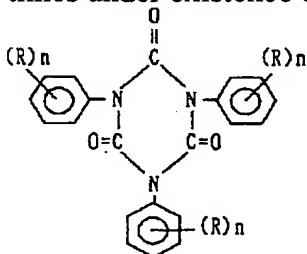
[Claim(s)]

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(-- R and n are the same as the above among a formula.) -- manufacturing method of triazine trione expressed.

[Claim 2] The manufacturing method of the triazine trione given in the 1st term of a claim given phase transfer catalyses are quaternary ammonium salt, phosphonate, or crown ether.

[Claim 3] The manufacturing method of the triazine trione given in the 2nd term of a claim given phase transfer catalyses are quaternary ammonium salt.

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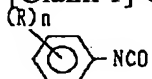
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CLAIMS

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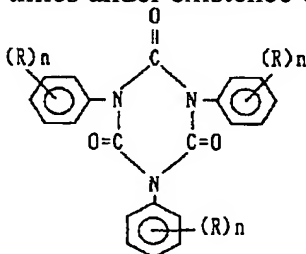
[Claim(s)]

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(-- R and n are the same as the above among a formula.) -- manufacturing method of triazine trione expressed.

[Claim 2] The manufacturing method of the triazine trione given in the 1st term of a claim given phase transfer catalyses are quarternary ammonium salt, phosphonate, or crown ether.

[Claim 3] The manufacturing method of the triazine trione given in the 2nd term of a claim given phase transfer catalyses are quarternary ammonium salt.

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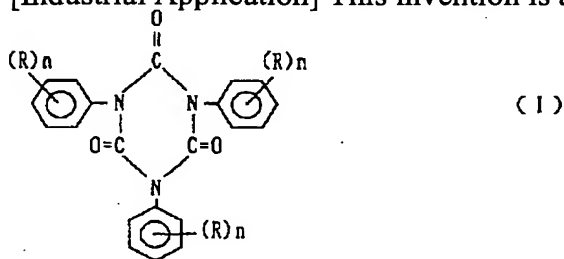
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## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention is a general formula (I).



the inside of a formula and R are the same -- or -- you may differ -- a halogen atom, a nitro group, and hydroxyl -- A low-grade alkyl group and low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, A low-grade alkylthio group and low-grade halo alkylthio group, a low-grade alkyl sulfonyl group, A low-grade halo alkyl sulfonyl group, a carboxy group, a low-grade alkoxy carbonyl group, Or you may differ. a low-grade alkylamino radical, a low-grade dialkylamino radical, and a phenyl group -- the same -- A halogen atom, low-grade alkyl group, and low-grade halo alkyl group, a lower alkoxy group, The phenyl group which has 1-5 substituents chosen from a low-grade haloalkoxy radical, low-grade alkylthio group, and low-grade halo alkylthio group, a phenoxy group -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The phenoxy group which has 1-5 substituents chosen from a low-grade halo alkylthio group, a phenylthio radical -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The phenylthio radical which has 1-5 substituents chosen from a low-grade halo alkylthio group, a phenyl sulfonyl group -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The phenyl sulfonyl group which has 1-5 substituents chosen from a low-grade halo alkylthio group, benzoyl -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The benzoyl which has 1-5 substituents chosen from a low-grade halo alkylthio group, an aralkyl radical -- or the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- the aralkyl radical which has 1-5 substituents chosen from a low-grade halo alkyl group, lower alkoxy group, low-grade haloalkoxy radical, low-grade alkylthio group, and low-grade halo alkylthio group is shown, and n shows the integer of 0-5. It is related with the manufacturing method of triazine trione expressed.

[0002]

[Description of the Prior Art] Various approaches are reported as the manufacture approach of triazine trione. For example, the approach of carrying out it 3 \*\*\*\*s, using cesium fluoride etc. as a catalyst (Beil.26,253, \*\* 26I, 76, \*\* 26III, 637 grade), The approach of carrying out them 3 \*\*\*\*s, using inorganic alkali salt, triethylamine, etc. as a catalyst (J. Am.Chem.Soc., 78, 4911-4914

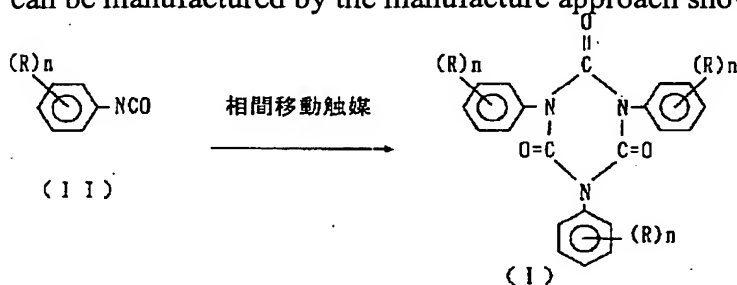
(1956)), How to make it into the bottom of existence of triethylamine 3 \*\*\*\*s under high pressure (the manufacture approaches, such as Bull.Chem.Soc.Jap., and 63, 3486 (1990), are reported.)

[0003]

[Problem(s) to be Solved by the Invention] The triazine trione which this invention person etc. is what found out this invention as a result of repeating research for the new manufacture approach of triazine trione wholeheartedly, this invention is the new reference manufacture approach of not indicating, and is expressed with a general formula (I) is compounds useful as intermediate fields, such as resin.

[0004]

[Means for Solving the Problem] General formula of this invention (I) The triazine trione expressed can be manufactured by the manufacture approach shown below.



(R and n are the same as the above among a formula.)

The triazine trione expressed with a general formula (I) can be manufactured by making into the bottom of existence of an inert solvent or un-existing, and existence of phase transfer catalysis 3 \*\*\*\*s of the isocyanic ester expressed with a general formula (II).

[0005] That what is necessary is just what does not check advance of the reaction of this invention remarkably in this invention as an inert solvent which can be used when using it although use of an inert solvent is not indispensable For example, aromatic hydrocarbon, such as benzene, toluene, and a xylene, an acetone, Ether, such as ketones, such as a cyclohexanone, ethyl ether, dioxane, and a tetrahydrofuran, Although inert solvents, such as aliphatic series amides, such as halogenated hydrocarbon, such as chloroform and a carbon tetrachloride, dimethylformamide, and dimethylacetamide, dimethylsulfoxide, and a tetrahydro sulfolane, can be used This invention is not limited to these inert solvents.

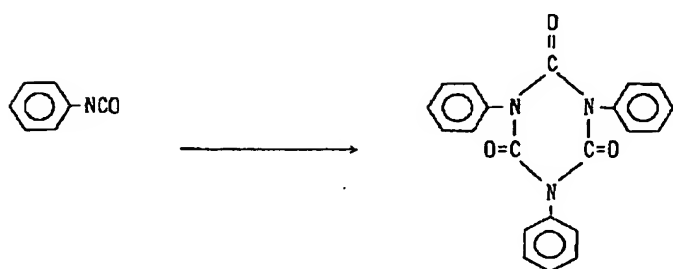
[0006] As phase transfer catalysis used by this invention, for example Tetrabutyl ammonium chloride, Tetrabutylammonium bromide, benzyl triethyl ammonium chloride, Benzyl triethyl ammonium bromide, trioctyl methylammonium chloride, Quarternary ammonium salt, such as trioctyl methylammonium bromide Tetrabutyl phosphonium chloride, tetrabutyl phosphonium bromide, Phosphonate, such as tetra-phenyl phosphonium chloride and tetra-phenyl phosphonium bromide It is the range of 0.01-0.1 mols preferably that what is necessary is to be able to use crown ether, and just to use the amount used in 0.001 mols - 0.5 mols to the isocyanic ester expressed with a general formula (II), choosing it suitably.

[0007] The range of the reaction temperature of this invention is 50 degrees C - 200 degrees C preferably that what is necessary is just to choose from the range of 0 degree C - 300 degrees C suitably. What is necessary is just to perform it in several minutes - 48 hours, although reaction time is not fixed with reaction temperature, a reaction scale, etc. The triazine trione expressed with a general formula (I) can be manufactured by isolating the specified substance with a conventional method after reaction termination.

[0008]

[Example] Although the typical example of this invention is illustrated below, this invention is not limited to these.

[0009] Example 1 1, 3, 5-triphenyl-perhydro-1,3,5-triazine - Manufacture of 2, 4, and 6-trione



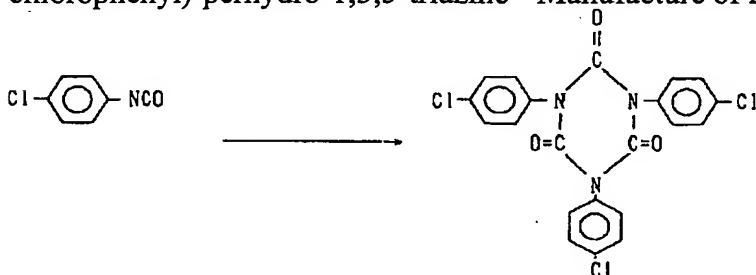
[0010] Heating stirring of 1 - 1. phenyl isocyanate 6g (0.05 mols) and the trioctyl methylammonium chloride 0.4g (5% mol) was carried out for 30 minutes at 150 degrees C. 4.9g of specified substance was obtained after reaction termination by carrying out ether washing of the solid-state crystal which cools reaction mixture and deposits, and drying. The obtained specified substance was in agreement with the preparation by IR (infrared absorption spectrum) analysis.

Physical properties m.p.> 265 degrees C 82% [0011] of yield 1-2. Heating stirring of phenyl isocyanate 6g (0.05 mols) and the trioctyl methylammonium chloride 0.1g (1.25% mol) was carried out for 30 minutes at 150 degrees C. 5.1g of specified substance was obtained after reaction termination by carrying out ether washing of the solid-state crystal which cools reaction mixture and deposits, and drying. The obtained specified substance was in agreement with the preparation by IR (infrared absorption spectrum) analysis.

Physical properties m.p.> 265 degrees C 85% [0012] of yield 1-3. Heating stirring of phenyl isocyanate 6g (0.05 mols) and the trioctyl methylammonium chloride 0.4g (1% mol) was carried out for 10 minutes at 200 degrees C. 4.9g of specified substance was obtained after reaction termination by carrying out ether washing of the solid-state crystal which cools reaction mixture and deposits, and drying. The obtained specified substance was in agreement with the preparation by IR (infrared absorption spectrum) analysis.

Physical properties m.p.> 265 degrees C 82% [0013] of yield 1-4. Heating stirring of phenyl isocyanate 6g (0.05 mols), tetrabutyl ammonium chloride 0.13g (5% mol), and the dimethylformamide was carried out for 30 minutes at 150 degrees C. 5.9g of specified substance was obtained after reaction termination by carrying out ether washing of the solid-state crystal which cools reaction mixture and deposits, and drying. The obtained specified substance was in agreement with the preparation by IR (infrared absorption spectrum) analysis.

Physical properties m.p.> 265 degrees C 98% [0014] of yield Example 2 1, 3, 5-Tori (p-chlorophenyl)-perhydro-1,3,5-triazine - Manufacture of 2, 4, and 6-trione



Heating stirring of p-chlorophenyl isocyanate 7.7g (0.05 mols) and the trioctyl ammonium chloride 0.4g (5% mol) was carried out for 30 minutes at 150 degrees C. 5.7g of specified substance was obtained after reaction termination by carrying out ether washing of the solid-state crystal which cools reaction mixture and deposits, and drying.

Physical properties m.p.> 265 degrees C 74% [0015] of yield It changed into the isocyanic ester which shows p-chlorophenyl isocyanate used in the example 3 example 2 by the following general formula (II), and other conditions were performed like the example 2. A result is shown in Table 1.

表 1

実施例	一般式(II)の(R) <sub>n</sub>	収率 (%)	物 性
3-1	p-Cl	49	m.p. 203.4℃
3-2	m-Cl	55	m.p. 215.4℃
3-3	p-CH <sub>3</sub>	81	m.p. 262.9℃
3-4	m-CH <sub>3</sub>	73	m.p. 199.6℃
3-5	o-CF <sub>3</sub>	16	m.p. 259.5℃
3-6	3,4-Cl <sub>2</sub>	64	m.p. >265℃

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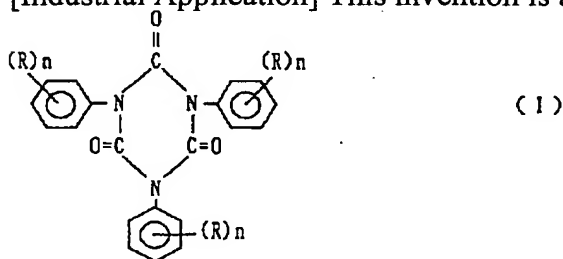
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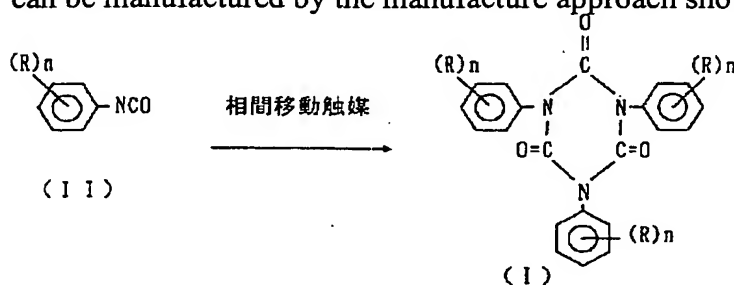
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[0005] That what is necessary is just what does not check advance of the reaction of this invention remarkably in this invention as an inert solvent which can be used when using it although use of an inert solvent is not indispensable For example, aromatic hydrocarbon, such as benzene, toluene, and a xylene, an acetone, Ether, such as ketones, such as a cyclohexanone, ethyl ether, dioxane, and a tetrahydrofuran, Although inert solvents, such as aliphatic series amides, such as halogenated hydrocarbon, such as chloroform and a carbon tetrachloride, dimethylformamide, and dimethylacetamide, dimethylsulfoxide, and a tetrahydro sulfolane, can be used This invention is not limited to these inert solvents.

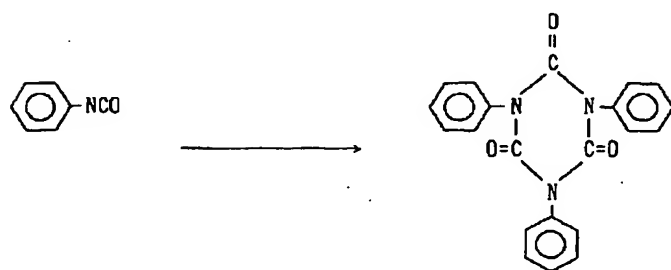
[0006] As phase transfer catalysis used by this invention, for example Tetrabutyl ammonium chloride, Tetrabutylammonium bromide, benzyl triethyl ammonium chloride, Benzyl triethyl ammonium bromide, trioctyl methylammonium chloride, Quarternary ammonium salt, such as trioctyl methylammonium bromide Tetrabutyl phosphonium chloride, tetrabutyl phosphonium bromide, Phosphonate, such as tetra-phenyl phosphonium chloride and tetra-phenyl phosphonium bromide It is the range of 0.01-0.1 mols preferably that what is necessary is to be able to use crown ether, and just to use the amount used in 0.001 mols - 0.5 mols to the isocyanic ester expressed with a general formula (II), choosing it suitably.

[0007] The range of the reaction temperature of this invention is 50 degrees C - 200 degrees C preferably that what is necessary is just to choose from the range of 0 degree C - 300 degrees C suitably. What is necessary is just to perform it in several minutes - 48 hours, although reaction time is not fixed with reaction temperature, a reaction scale, etc. The triazine trione expressed with a general formula (I) can be manufactured by isolating the specified substance with a conventional method after reaction termination.

[0008]

[Example] Although the typical example of this invention is illustrated below, this invention is not limited to these.

[0009] Example 1 1, 3, 5-triphenyl-perhydro-1,3,5-triazine - Manufacture of 2, 4, and 6-trione



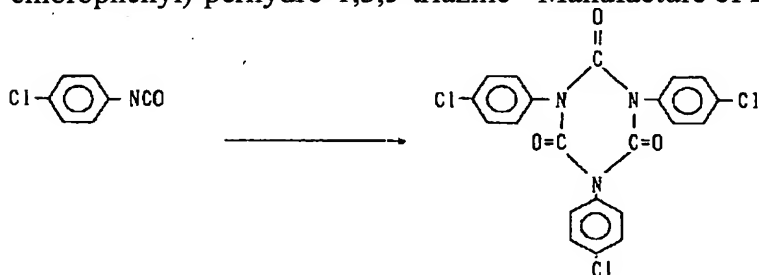
[0010] Heating stirring of 1 - 1. phenyl isocyanate 6g (0.05 mols) and the trioctyl methylammonium chloride 0.4g (5% mol) was carried out for 30 minutes at 150 degrees C. 4.9g of specified substance was obtained after reaction termination by carrying out ether washing of the solid-state crystal which cools reaction mixture and deposits, and drying. The obtained specified substance was in agreement with the preparation by IR (infrared absorption spectrum) analysis.

Physical properties m.p.> 265 degrees C 82% [0011] of yield 1-2. Heating stirring of phenyl isocyanate 6g (0.05 mols) and the trioctyl methylammonium chloride 0.1g (1.25% mol) was carried out for 30 minutes at 150 degrees C. 5.1g of specified substance was obtained after reaction termination by carrying out ether washing of the solid-state crystal which cools reaction mixture and deposits, and drying. The obtained specified substance was in agreement with the preparation by IR (infrared absorption spectrum) analysis.

Physical properties m.p.> 265 degrees C 85% [0012] of yield 1-3. Heating stirring of phenyl isocyanate 6g (0.05 mols) and the trioctyl methylammonium chloride 0.4g (1% mol) was carried out for 10 minutes at 200 degrees C. 4.9g of specified substance was obtained after reaction termination by carrying out ether washing of the solid-state crystal which cools reaction mixture and deposits, and drying. The obtained specified substance was in agreement with the preparation by IR (infrared absorption spectrum) analysis.

Physical properties m.p.> 265 degrees C 82% [0013] of yield 1-4. Heating stirring of phenyl isocyanate 6g (0.05 mols), tetrabutyl ammonium chloride 0.13g (5% mol), and the dimethylformamide was carried out for 30 minutes at 150 degrees C. 5.9g of specified substance was obtained after reaction termination by carrying out ether washing of the solid-state crystal which cools reaction mixture and deposits, and drying. The obtained specified substance was in agreement with the preparation by IR (infrared absorption spectrum) analysis.

Physical properties m.p.> 265 degrees C 98% [0014] of yield Example 2 1, 3, 5-Tri (p-chlorophenyl)-perhydro-1,3,5-triazine - Manufacture of 2, 4, and 6-trione



Heating stirring of p-chlorophenyl isocyanate 7.7g (0.05 mols) and the trioctyl ammonium chloride 0.4g (5% mol) was carried out for 30 minutes at 150 degrees C. 5.7g of specified substance was obtained after reaction termination by carrying out ether washing of the solid-state crystal which cools reaction mixture and deposits, and drying.

Physical properties m.p.> 265 degrees C 74% [0015] of yield It changed into the isocyanic ester which shows p-chlorophenyl isocyanate used in the example 3 example 2 by the following general formula (II), and other conditions were performed like the example 2. A result is shown in Table 1.

表 1

実施例	一般式(II)の(R) <sub>n</sub>	収率 (%)	物 性
3-1	p-Cl	49	m.p. 203.4℃
3-2	m-Cl	55	m.p. 215.4℃
3-3	p-CH <sub>3</sub>	81	m.p. 262.9℃
3-4	m-CH <sub>3</sub>	73	m.p. 199.6℃
3-5	o-CF <sub>3</sub>	16	m.p. 259.5℃
3-6	3,4-Cl <sub>2</sub>	64	m.p. >265℃

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[Translation done.]



## \* NOTICES \*

JPO and NCIPi are not responsible for any damages caused by the use of this translation.

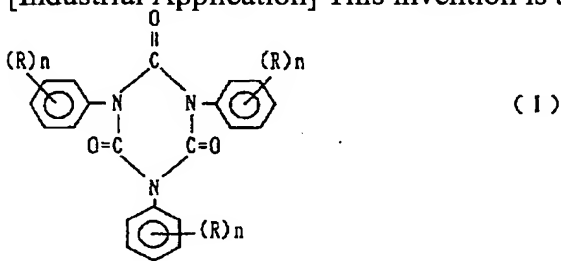
- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention is a general formula (I).



the inside of a formula and R are the same -- or -- you may differ -- a halogen atom, a nitro group, and hydroxyl -- A low-grade alkyl group and low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, A low-grade alkylthio group and low-grade halo alkylthio group, a low-grade alkyl sulfonyl group, A low-grade halo alkyl sulfonyl group, a carboxy group, a low-grade alkoxy carbonyl group, Or you may differ. a low-grade alkylamino radical, a low-grade dialkylamino radical, and a phenyl group -- the same -- A halogen atom, low-grade alkyl group, and low-grade halo alkyl group, a lower alkoxy group, The phenyl group which has 1-5 substituents chosen from a low-grade haloalkoxy radical, low-grade alkylthio group, and low-grade halo alkylthio group, a phenoxy group -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The phenoxy group which has 1-5 substituents chosen from a low-grade halo alkylthio group, a phenylthio radical -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The phenylthio radical which has 1-5 substituents chosen from a low-grade halo alkylthio group, a phenyl sulfonyl group -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The phenyl sulfonyl group which has 1-5 substituents chosen from a low-grade halo alkylthio group, benzoyl -- the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- A low-grade halo alkyl group, a lower alkoxy group, a low-grade haloalkoxy radical, a low-grade alkylthio group, The benzoyl which has 1-5 substituents chosen from a low-grade halo alkylthio group, an aralkyl radical -- or the same -- or -- you may differ -- a halogen atom and a low-grade alkyl group -- the aralkyl radical which has 1-5 substituents chosen from a low-grade halo alkyl group, lower alkoxy group, low-grade haloalkoxy radical, low-grade alkylthio group, and low-grade halo alkylthio group is shown, and n shows the integer of 0-5. It is related with the manufacturing method of triazine trione expressed.

[0002]

[Description of the Prior Art] Various approaches are reported as the manufacture approach of triazine trione. For example, the approach of carrying out it 3 \*\*\*\*s, using cesium fluoride etc. as a catalyst (Beil.26,253, \*\* 26I, 76, \*\* 26III, 637 grade), The approach of carrying out them 3 \*\*\*\*s, using inorganic alkali salt, triethylamine, etc. as a catalyst (J. Am.Chem.Soc., 78, 4911-4914

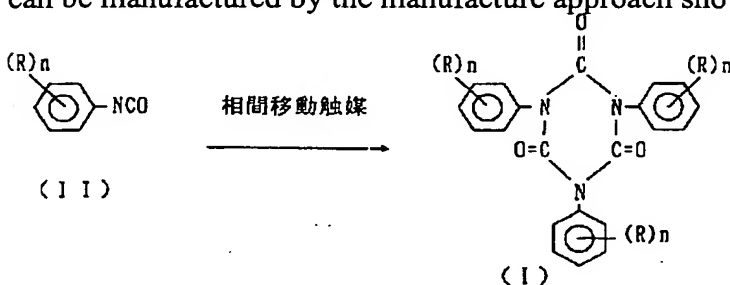
(1956)), How to make it into the bottom of existence of triethylamine 3 \*\*\*\*s under high pressure (the manufacture approaches, such as Bull.Chem.Soc.Jap., and 63, 3486 (1990), are reported.)

[0003]

[Problem(s) to be Solved by the Invention] The triazine trione which this invention person etc. is what found out this invention as a result of repeating research for the new manufacture approach of triazine trione wholeheartedly, this invention is the new reference manufacture approach of not indicating, and is expressed with a general formula (I) is compounds useful as intermediate fields, such as resin.

[0004]

[Means for Solving the Problem] General formula of this invention (I) The triazine trione expressed can be manufactured by the manufacture approach shown below.



(R and n are the same as the above among a formula.)

The triazine trione expressed with a general formula (I) can be manufactured by making into the bottom of existence of an inert solvent or un-existing, and existence of phase transfer catalysis 3 \*\*\*\*s of the isocyanic ester expressed with a general formula (II).

[0005] That what is necessary is just what does not check advance of the reaction of this invention remarkably in this invention as an inert solvent which can be used when using it although use of an inert solvent is not indispensable For example, aromatic hydrocarbon, such as benzene, toluene, and a xylene, an acetone, Ether, such as ketones, such as a cyclohexanone, ethyl ether, dioxane, and a tetrahydrofuran, Although inert solvents, such as aliphatic series amides, such as halogenated hydrocarbon, such as chloroform and a carbon tetrachloride, dimethylformamide, and dimethylacetamide, dimethylsulfoxide, and a tetrahydro sulfolane, can be used This invention is not limited to these inert solvents.

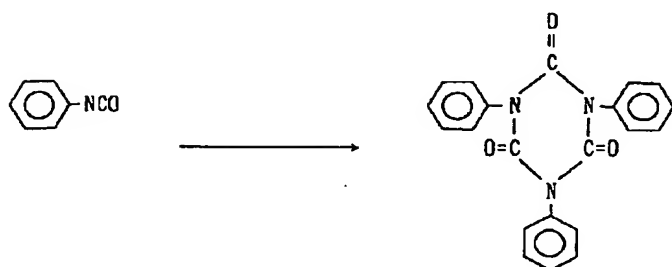
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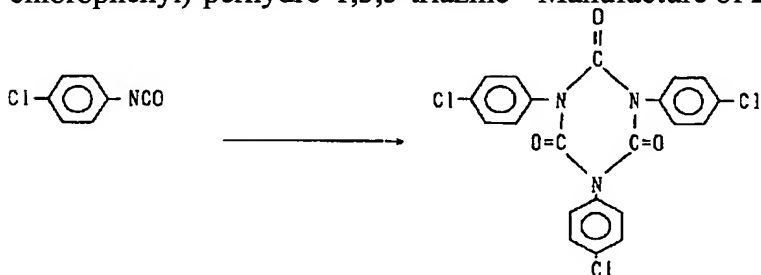
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[Translation done.]

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(12) 公開特許公報 (A)

(11)特許出願公開番号

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(51)Int.Cl. <sup>5</sup>	識別記号	庁内整理番号	F I	技術表示箇所
C 0 7 D 251/34		A		
B 0 1 J 31/02				
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// C 0 7 B 61/00	3 0 0			

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(21)出願番号 特願平4-86034

(22)出願日 平成4年(1992)3月9日

(71)出願人 000232623

日本農薬株式会社

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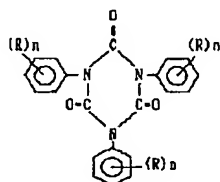
(74)代理人 弁理士 苧 経夫 (外2名)

(54)【発明の名称】 トリアジントリオン類の製造法

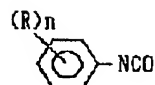
(57)【要約】 (修正有)

【目的】 樹脂類の中間体として有用なトリアジントリオン類の新規な製造方法を提供する。

【構成】 一般式(II)で表されるイソシアン酸エステル類を相間移動触媒の存在下に三量化することを特徴とする一般式(I)で表されるトリアジントリオン類の製造法。



(I)

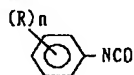


(II)

(式中、Rは同一又は異ってもよく、ハロゲン原子、N O<sub>2</sub>、OH、低級アルキル基、低級アルコキシ基、低級アルコシカルボニル基等を表し、nは0-2の整数である)。

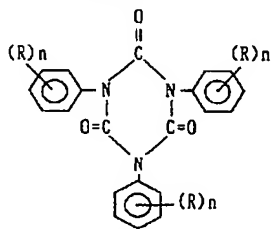
【特許請求の範囲】

【請求項1】 一般式(11)



(11)

(式中、Rは同一又は異なっても良く、ハロゲン原子、ニトロ基、ヒドロキシル基、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基、低級アルキルスルホニル基、低級ハロアルキルスルホニル基、カルボキシ基、低級アルコキシカルボニル基、低級アルキルアミノ基、低級ジアルキルアミノ基、フェニル基、同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するフェニル基、フェノキシ基、同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するフェノキシ基、フェニルチオ基、同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するフェニルチオ基、フェニルスルホニル基、同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するフェニルスルホニル基、ベンゾイル基、同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するベンゾイル基、アラルキル基又は同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するアラルキル基を示し、nは0～5の整数を示す。)で表されるイソシアヌ酸エステル類を相間移動触媒の存在下に三量化することを特徴とする一般式(1)



(1)

(式中、R及びnは前記に同じ。)で表されるトリアジ

ントリオン類の製造法。

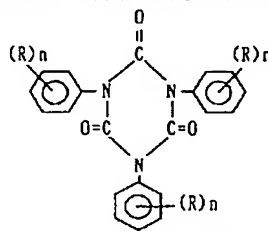
【請求項2】 相間移動触媒が四級アンモニウム塩類、ホスホン酸塩類又はクラウンエーテル類である請求項第1項記載のトリアジントリオン類の製造法。

【請求項3】 相間移動触媒が四級アンモニウム塩類である請求項第2項記載のトリアジントリオン類の製造法。

【発明の詳細な説明】

【0001】

10 【産業上の利用分野】本発明は一般式(1)



(1)

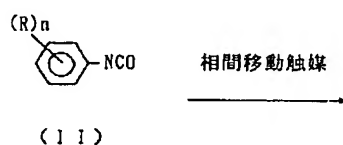
(式中、Rは同一又は異なっても良く、ハロゲン原子、ニトロ基、ヒドロキシル基、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基、低級アルキルスルホニル基、低級ハロアルキルスルホニル基、カルボキシ基、低級アルコキシカルボニル基、低級アルキルアミノ基、低級ジアルキルアミノ基、フェニル基、同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するフェニル基、フェノキシ基、同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するフェノキシ基、フェニルチオ基、同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するフェニルチオ基、フェニルスルホニル基、同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するフェニルスルホニル基、ベンゾイル基、同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するベンゾイル基、アラルキル基又は同一又は異なっても良く、ハロゲン原子、低級アルキル基、低級ハロアルキル基、低級アルコキシ基、低級ハロアルコキシ基、低級アルキルチオ基、低級ハロアルキルチオ基から選択される1～5個の置換基を有するアラルキル基を示し、nは0～5の整数を示す。)で表されるトリアジ

50

される1~5個の置換基を有するアラルキル基を示し、  
nは0~5の整数を示す。)で表されるトリアジントリ  
オン類の製造法に関するものである。

【0002】

【従来技術】トリアジントリオン類の製造方法としては  
種々の方法が報告されており、例えばフッ化セシウム等  
を触媒として三量化させる方法(Beil. 26, 25  
3, 同 26I, 76, 同 26III, 637等)、無  
機アルカリ塩、トリエチルアミン等を触媒として三量化  
させる方法(J. Am. Chem. Soc., 78, 4 10  
911-4914 (1956))、トリエチルアミンの  
存在下に高圧下に三量化させる方法(Bull. Che  
m. Soc. Jap., 63, 3486 (1990))等\*



(式中、R及びnは前記に同じ。)

一般式(11)で表されるイソシアネートエステル類を不  
活性溶媒の存在下又は不存在下及び相間移動触媒の存在  
下に三量化させることにより一般式(1)で表されるト  
リアジントリオン類を製造することができる。

【0005】本発明では不活性溶媒の使用は必須ではな  
いが、使用する場合に使用できる不活性溶媒としては本  
発明の反応の進行を著しく阻害しないものであれば良  
く、例えばベンゼン、トルエン、キシレン等の芳香族炭  
化水素類、アセトン、シクロヘキサノン等のケトン類、  
エチルエーテル、ジオキサン、テトラヒドロフラン等の  
エーテル類、クロロホルム、四塩化炭素等のハロゲン化  
炭化水素類、ジメチルホルムアミド、ジメチルアセトア  
ミド等の脂肪族アミド類、ジメチルスルホキサイド、テ  
トラヒドロスルホラン等の不活性溶媒を使用することが  
できるが、本発明はこれらの不活性溶媒に限定されるも  
のではない。

【0006】本発明で使用する相間移動触媒としては、  
例えばテトラブチルアンモニウムクロリド、テトラブチ  
ルアンモニウムブロミド、ベンジルトリエチルアンモニ  
ウムクロリド、ベンジルトリエチルアンモニウムブロミ  
ド、トリオクチルメチルアンモニウムクロリド、トリオ※

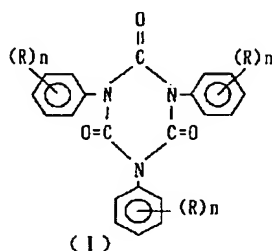
\*の製造方法が報告されている。

【0003】

【発明が解決しようとする課題】本発明者等はトリアジ  
ントリオン類の新規な製造方法を鋭意研究を重ねた結  
果、本発明を見出したもので、本発明は文献未記載の新  
規な製造方法であり、一般式(1)で表されるトリアジ  
ントリオン類は樹脂等の中間体として有用な化合物であ  
る。

【0004】

【課題を解決するための手段】本発明の一般式(I)で表  
されるトリアジントリオン類は、下記に示す製造方法に  
より製造することができる。



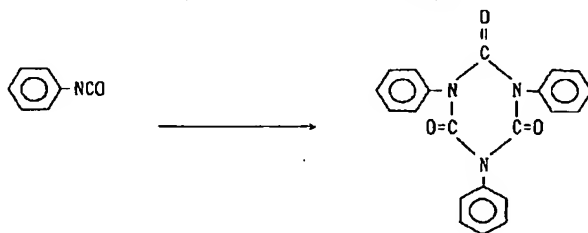
※クチルメチルアンモニウムブロミド等の四級アンモニウ  
ム塩類、テトラブチルホスホニウムクロリド、テトラブ  
チルホスホニウムブロミド、テトラフェニルホスホニウ  
ムクロリド、テトラフェニルホスホニウムブロミド等の  
ホスホン酸塩類、クラウンエーテル類等を使用すること  
ができ、その使用量は一般式(11)で表されるイソシ  
アネートエステル類に対して0.001モル~0.5モル  
の範囲で適宜選択して使用すれば良く、好ましくは0.  
01~0.1モルの範囲である。

【0007】本発明の反応温度は0℃~300℃の範囲  
から適宜選択すれば良く、好ましくは50℃~200℃  
の範囲である。反応時間は反応温度、反応規模等により  
一定しないが数分~48時間の範囲でおこなえば良い。  
反応終了後、目的物を常法により単離することにより一  
般式(1)で表されるトリアジントリオン類を製造する  
ことができる。

【0008】

【実施例】以下に本発明の代表的な実施例を例示する  
が、本発明はこれらに限定されるものではない。

【0009】実施例1 1, 3, 5-トリフェニル-  
ペルヒドロ-1, 3, 5-トリアジン-2, 4, 6-トリ  
オンの製造



5

【0010】1-1. フェニルイソシアナート6g (0.05モル)、トリオクチルメチルアンモニウムクロリド0.4g (5%モル)を150℃で30分間加熱攪拌した。反応終了後、反応液を冷却し析出する固体結晶をエーテル洗浄し乾燥することにより目的物4.9gを得た。得られた目的物はIR (赤外吸収スペクトル) 分析で標品と一致した。

物性 m. p. >265℃ 収率82%

【0011】1-2. フェニルイソシアナート6g (0.05モル)、トリオクチルメチルアンモニウムクロリド0.1g (1.25%モル)を150℃で30分間加熱攪拌した。反応終了後、反応液を冷却し析出する固体結晶をエーテル洗浄し乾燥することにより目的物5.1gを得た。得られた目的物はIR (赤外吸収スペクトル) 分析で標品と一致した。

物性 m. p. >265℃ 収率85%

【0012】1-3. フェニルイソシアナート6g (0.05モル)、トリオクチルメチルアンモニウムク\*

6

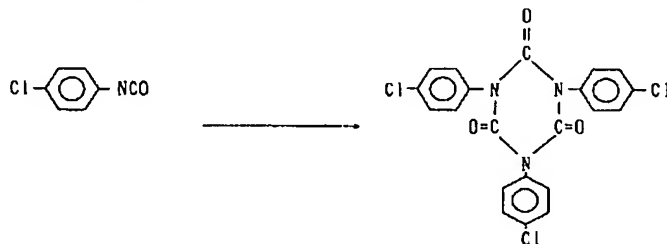
\*ロリド0.4g (1%モル)を200℃で10分間加熱攪拌した。反応終了後、反応液を冷却し析出する固体結晶をエーテル洗浄し乾燥することにより目的物4.9gを得た。得られた目的物はIR (赤外吸収スペクトル) 分析で標品と一致した。

物性 m. p. >265℃ 収率82%

【0013】1-4. フェニルイソシアナート6g (0.05モル)、テトラブチルアンモニウムクロリド0.13g (5%モル)及びジメチルホルムアミドを150℃で30分間加熱攪拌した。反応終了後、反応液を冷却し析出する固体結晶をエーテル洗浄し乾燥することにより目的物5.9gを得た。得られた目的物はIR (赤外吸収スペクトル) 分析で標品と一致した。

物性 m. p. >265℃ 収率98%

【0014】実施例2 1, 3, 5-トリ(p-クロロフェニル)-ペルヒドロ-1, 3, 5-トリアジン-2, 4, 6-トリオンの製造



p-クロロフェニルイソシアナート7.7g (0.05モル)、トリオクチルアンモニウムクロリド0.4g (5%モル)を150℃で30分間加熱攪拌した。反応終了後、反応液を冷却し析出する固体結晶をエーテル洗浄し乾燥することにより目的物5.7gを得た。

物性 m. p. >265℃ 収率74%

※

※【0015】実施例3

実施例2で使用したp-クロロフェニルイソシアナートを下記一般式(II)で示すイソシアネート類に変え、他の条件は実施例2と同様にして行った。結果を表1に示す。

表1

実施例	一般式(II)の(R) <sub>n</sub>	収率 (%)	物 性
3-1	p-Cl	49	m. p. 203.4℃
3-2	m-Cl	55	m. p. 215.4℃
3-3	p-CH <sub>3</sub>	81	m. p. 262.9℃
3-4	m-CH <sub>3</sub>	73	m. p. 199.6℃
3-5	o-CF <sub>3</sub>	16	m. p. 259.5℃
3-6	3, 4-Cl <sub>2</sub>	64	m. p. >265℃